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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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10/088,738

07/23/2002

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EXAMINER

SINGH, PREM C

ART UNIT

PAPER NUMBER

1797

MAIL DATE

DELIVERY MODE

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PAPER

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

<b>Office Action Summary</b>	<b>Application No.</b> 10/088,738	<b>Applicant(s)</b> HUMBLLOT ET AL.	
	<b>Examiner</b> PREM C. SINGH	<b>Art Unit</b> 1797	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

### Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

### Status

- 1) ☒ Responsive to communication(s) filed on 02 September 2008.
- 2a) ☒ This action is **FINAL**.                      2b) ☐ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

### Disposition of Claims

- 4) ☒ Claim(s) 1-6, 11-18 and 20-29 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-6, 11-18 and 20-29 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

### Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

### Priority under 35 U.S.C. § 119

- 12) ☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☒ All    b) ☐ Some \*    c) ☐ None of:
1. ☒ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

### Attachment(s)

- |  |   |
|--|---|
| 1) <input type="checkbox"/> Notice of References Cited (PTO-892)                     | 4) <input type="checkbox"/> Interview Summary (PTO-413)           |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948) | Paper No(s)/Mail Date. _____                                      |
| 3) <input type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08)          | 5) <input type="checkbox"/> Notice of Informal Patent Application |
| Paper No(s)/Mail Date _____  | 6) <input type="checkbox"/> Other: _____                          |

## **DETAILED ACTION**

### ***Response to Amendment***

1. Amendment to claims 1 and 6, cancellation of claims 7-10 and 19, and addition of new claims 25-29 is noted.
2. Rejection of claim 1 under 35 U.S.C. 112, first paragraph, is withdrawn.
3. New grounds of rejection necessitated by amendment to the claims and addition of new claims follow.

### ***Claim Rejections - 35 USC § 103***

4. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(e), (f) or (g) prior art under 35 U.S.C. 103(a).

5. Claims 1-6, 11-18 and 20-29 are rejected under 35 U.S.C. 103(a) as being unpatentable over Zimmermann et al (US Patent 5,849,176) in view of Reed et al (US Patent 5,656,150).

6. With respect to claims 1-4, 6, and 25-29, Zimmermann relates to the protection of tubular reactors or heat exchangers against coke formation in plants for converting hydrocarbons and other organic compounds at high temperatures in the gaseous phase (See abstract). Zimmermann further discloses pre-treatment of chrome-nickel steel with trimethyl-silyl-methyl mercaptan for 60 minutes at 880°C (See column 5, lines 49-54) and adds, "In place of compounds that simultaneously contain both silicon and sulfur, mixtures of silicon compounds and sulfur compounds also attain the same effect as coke formation inhibitors." (Column 6, lines 52-57). Zimmermann further discloses "The

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compound containing silicon and/or sulfur is preferably selected from the group that consists of dimethyl sulfide, tetramethyl silane, and their mixtures. However, other volatile compounds can also be used.” (Column 2, lines 65-67; column 3, lines 1-3). It is to be noted that Zimmermann discloses use of dimethylsulfide (DMS) in the surface pretreatment (See column 2, lines 65-67; column 3, lines 1-3) and uses dimethyldisulfide (DMDS) as coke inhibitor by adding in the hydrocarbon feed to be cracked (See column 4, lines 3-7). Thus, it would have been obvious to one skilled in the art at the time of invention to modify Zimmerman invention and use dimethyldisulfide (DMDS) to pretreat the surface because DMS and DMDS are both inhibitors of coke and one skilled in the art could use either of the two. See *In Re Ruff*, 256 F.2d 590, 118 USPQ 340 (CCPA 1958) and *In Re Fout*, 675 F.2d 297, 213 USPQ 532 (CCPA 1982).

Zimmermann does not specifically disclose using hexamethyldisiloxane.

Reed discloses a novel method similar to Zimmermann for treating the radiant tubes of a fired pyrolysis heater with an antifoulant composition for inhibiting coke deposition. Reed uses several silicon compounds including hexamethyldisiloxane (See column 4, lines 32-62).

Since Zimmermann and Reed both inventions disclose coke inhibition on the inner tubes of a cracking reactor by using silicon compounds, it would have been obvious to one skilled in the art at the time of invention to modify Zimmermann invention and use hexamethyldisiloxane for coke inhibition as disclosed by Reed because this composition is also effective as other compositions disclosed by Zimmermann. See *In*

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*Re Ruff*, 256 F.2d 590, 118 USPQ 340 (CCPA 1958) and *In Re Fout*, 675 F.2d 297, 213 USPQ 532 (CCPA 1982).

Zimmermann uses a mixture of hydrogen and methane during pre-treatment (see column 5, lines 51-53) but does not specifically disclose using steam.

Reed discloses use of steam in the pretreatment of radiant tubes (See column 6, lines 5-15). Thus, it would have been obvious to one skilled in the art at the time the invention was made to modify Zimmermann invention and use steam in place of hydrogen/methane mixture and make the treatment process more economical because use of steam in place of hydrogen/methane mixture will be cheaper.

Zimmermann uses Si:S atomic ratio between 1:1 to 5:1 (See column 3, lines 45-50), a temperature of 880°C and a time of 60 minutes for pretreatment which lie between the claimed temperature and time ranges. In the case where the claimed ranges “overlap or lie inside ranges disclosed by the prior art” a *prima facie* case of obviousness exists. See *In re Wertheim*, 541 F.2d 257, 191 USPQ 90 (CCPA 1976); *In re Woodruff*, 919 F.2d 1575, 16 USPQ2d 1934 (Fed. Cir. 1990).

Zimmermann also discloses concentration of tri-methyl-silyl-methyl-mercaptan in Experiments 5-9, but does not specifically disclose concentration of silicon. It would have been obvious to one skilled in the art at the time the invention was made to modify Zimmermann invention and specify silicon concentration for proper control of coking in the tubes.

Zimmermann invention does not specifically disclose the inhibition of coke percentage on the metal walls, however, the invention does disclose coke formation

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rates in different experiments (See Examples 5 and 6 and figures 5 and 6).

Zimmermann also discloses figure 7 showing results of n-heptane cracking. The figure shows that by adding tri-methyl-silyl-ethyl-mercaptan to the feed the coking rate is reduced from about 140 to about 5  $\mu\text{g}/\text{cm}^2 \text{ min}$  (96% reduction). It is expected that by treating the metal walls with the inhibiting compositions of Zimmermann and Reed also, the coking will be reduced in a similar range. Thus, it would have been obvious to one skilled in the art at the time of invention to modify Zimmermann invention and determine the inhibition of coke percentage, which is expected to be in a range, including as claimed, because Zimmermann in view of Reed is using hexamethyldisiloxane and DMS/DMDS for treating the metal walls under similar operating conditions as claimed by the Applicant.

7. With respect to claim 5, Zimmermann discloses, "Figures 1-7 show in respect to pre-activated samples of chrome-nickel steel and samples that display a reduced coking tendency due to special thermal pre-treatment with compounds containing silicon and sulfur, the dependency of coke formation rates on test time during the pyrolysis of n-heptane in nitrogen and in steam as the diluent when known coke formation inhibitors and inhibitors according to the invention are added." (Column 3, lines 54-63).

Although Zimmermann uses nitrogen and steam as diluent separately, it would have been obvious to one skilled in the art at the time the invention was made to modify Zimmermann invention and use a mixture of steam and nitrogen as diluent because the

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mixture is also expected to be effective for coke inhibition due to the fact that the use of steam and nitrogen individually, is effective. See *In Re Kerkhoven*, 626 F.2d 846, 850, 205 USPQ 1069, 1072 (CCPA 1980).

8. With respect to claim 15, Reed uses several silicon compounds including hexamethyldisiloxane (See column 4, lines 32-62).

9. With respect to claim 11, 16-18, 20, 22-24, Zimmermann discloses "In all cases the atomic ratio of silicon and sulfur is between 5:1 to 1:1". (Column 2, lines 48-49). Zimmermann also discloses using 20 to 1000 ppm of the additive composition (See column 2, lines 29-31).

10. With respect to claims 12 and 21, Zimmermann discloses using normal pressure (1 atm pressure = 1.013 bar) (See column 4, lines 47-48).

11. With respect to claims 13 and 14, Zimmermann discloses, "On a pre-activated sample of chrome nickel steel dependence of coke formation rate on the test time during n-heptane pyrolysis without and with the addition of 85 ppm dimethyl disulfide is shown in figure 2." (Column 4, lines 3-7).



***Response to Arguments***

12. Applicant's arguments filed 09/02/2008 have been fully considered but they are not persuasive.

13. The Applicant argues,

“Zimmerman discloses a process consisting in adding compounds containing silicon and sulfur ('additive compositions') to a feed to be cracked. The process of Zimmerman is not a pretreatment of the metal surfaces of a reactor with a stream of steam comprising hexamethyldisiloxane and DMDS. Moreover, the stream of steam disclosed in Zimmerman is only used as a diluent for the additive compositions of Zimmerman added to the feed during pyrolysis of n-heptane, as specified for example in column 2 line 29, example 9 and table 2 of Zimmerman. The comparative example 5 of Zimmerman is not an example according to the invention of Zimmerman, and gives lower results (higher coke formation rates) than those of example 9, which is an example according to the invention in Zimmerman ' 176 (compare figures 5 and 6). Therefore, the person skilled in the art would not have been drawn to select this example 5 and would not modify this example by adding all the characteristics of the present invention as claimed in amended claim 1. (See also the discussion below regarding new claim 25 and the deficiencies in Zimmermann). Moreover, the Si:S ratio in Zimmerman is between 5:1 and 1:1, and fails to disclose the range of about 2:1 and 1:2 currently found in amended claim 1. As to Reed, this reference fails to remedy the deficiencies described above for Zimmermann. Reed discloses an antifoulant composition comprising tin and silicon (column 4 lines 15-20). Reed neither disclose nor suggest a pretreatment with a stream of steam comprising both hexamethyldisiloxane and at least one non-silicon-containing sulphur. (See also the discussion below regarding new claim 25 and Reed). Therefore, Reed also does not disclose the claimed ratio let alone any Si:S ratio. In conclusion, it is maintained that the recited documents (even in combination) do not suggest the invention of claim 1 as presently claimed.

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Consequently, it is respectfully submitted that the presently claimed invention of claim 1 is new and satisfies the condition of non-obviousness as does the claim 1 dependant claims”.

The Applicant's argument is not persuasive because Zimmermann uses coke inhibiting compositions similar to the Applicant's claim in both ways: in treating the metal walls and also in blending with the cracking feedstock (See Examples 5 and 6).

Examples 5 and 6 and figures 5 and 6 clearly show that when the metal walls are treated with tri-methyl-silyl-ethyl-mercaptan (TMSMM), the coke formation rate is about  $3.5 \mu\text{g}/\text{cm}^2 \text{ min}$  (figure 5) and about  $2.5 \mu\text{g}/\text{cm}^2 \text{ min}$  (figure 6) when the TMSMM is added to the feed passing through the pretreated tube. Thus, one skilled in the art would use the coke inhibiting composition either for pretreating the metal walls or would also add the inhibiting composition in the cracking feed for enhanced inhibition.

Zimmermann in view of Reed discloses metal wall treatment with hexamethyldisiloxane and DMDS in presence of steam (See Office action above under claim 1).

Zimmermann's range of Si:S ratio overlaps the claimed range. In the case where the claimed ranges “overlap or lie inside ranges disclosed by the prior art” a *prima facie* case of obviousness exists. See *In re Wertheim*, 541 F.2d 257, 191 USPQ 90 (CCPA 1976); *In re Woodruff*, 919 F.2d 1575, 16 USPQ2d 1934 (Fed. Cir. 1990). In conclusion, claims 1 and 25 and their dependent claims are prima facie obvious over Zimmermann in view of Reed.

14. The Applicant argues,

“In addition, a second Zimmermann reference (WO95/22588 and associated US 5922192) sheds light on how Example 5 and 9 of Zimmermann ' 176 should be considered. Particularly, Example 6 of WO

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95/22588 discloses that Steam is not suitable for long-lasting suppression of coking on materials pretreated with tri-methyl-silyl-methyl-mercaptan. See col. 7, lines 44-61 of the corresponding US Patent No. 5922192 quoted below.....".

The Applicant's argument is moot because the above cited reference is not a part of the rejection.

15. The Applicant argues,

Reed '150 was applied as a secondary reference for discussing steam in the pretreatment step. For the reasons outlined above, it is respectfully submitted that Reed fails to remedy the above noted deficiencies in Zimmermann ' 176 as the disclosure in Reed does not warrant the revisions asserted in the Office Action when considering the combination of features in claim 1 and the teaching of avoiding steam in the context of the base reference of Zimmermann ' 176".

The Applicant's argument is not persuasive because Reed reference has been used only to show that hexamethyldisiloxane is one of the inhibiting compositions similar to the ones disclosed by Zimmermann, and steam could be used in place of mixture of hydrogen and methane as disclosed by Zimmermann. Thus, there are no deficiencies in Zimmermann to be cured by Reed.

16. The Applicant argues,

Furthermore, new independent claim 25 includes additional language that further differentiates the claim 25 invention over either or both of Zimmermann and Reed, in any combination. For instance, in connection with the noted process steps set out in claim 25, the claim 25 invention provides for a high inhibition level of coke as in the range of 36% to 66%, which is a level not attributable to the relied upon references due to the above noted differences. Support for this language in claim 25 is found, for

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example, in the paragraph: bridging pages 18 and 19 and on page 12, line 14. Further, new claim 26 describes an inhibition of coke percentage on the metal walls of the reactor and the heat exchanger from 53% to 66%. (See also pages 18 and 19). Also, dependent claim 29 reference temperature values for the process not disclosed or suggested for the noted claim 25 process. The temperature range for the reactor in Reed '150 is between 537.77° C to 1093° C (1000° F to 2000° F). See col. 6, lines 5-22".

The Applicant's argument is not persuasive because new claim 25 has been addressed in the Office action above. Zimmermann discloses coke formation rates in different experiments (See Examples 5 and 6 and figures 5 and 6). Zimmermann also discloses figure 7 showing results of n-heptane cracking. The figure shows that by adding tri-methyl-silyl-ethyl-mercaptan to the feed the coking rate is reduced from 140 to 5 µg/cm<sup>2</sup> min (96% reduction). It is expected that by treating the metal walls with the inhibiting compositions of Zimmermann and Reed also, the coking will be reduced in a similar range. Dependent claim 29 claims a temperature range of 750 to 850°C and Reed discloses a temperature range of 537.7 to 1093°C which overlaps the claimed temperature range. In the case where the claimed ranges "overlap or lie inside ranges disclosed by the prior art" a *prima facie* case of obviousness exists. See *In re Wertheim*, 541 F.2d 257, 191 USPQ 90 (CCPA 1976); *In re Woodruff*, 919 F.2d 1575, 16 USPQ2d 1934 (Fed. Cir. 1990).

In conclusion, the claimed invention is *prima facie* obvious over Zimmermann in view of Reed.

***Conclusion***

17. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Prem C. Singh whose telephone number is 571-272-6381. The examiner can normally be reached on MF 7:00 AM-3:30 PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Glenn Caldarola can be reached on 571-272-1444. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

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PS 110308

/Glenn A Caldarola/  
Acting SPE of Art Unit 1797

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